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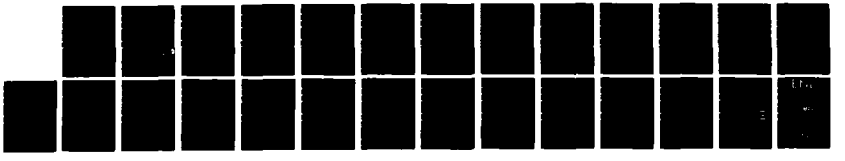

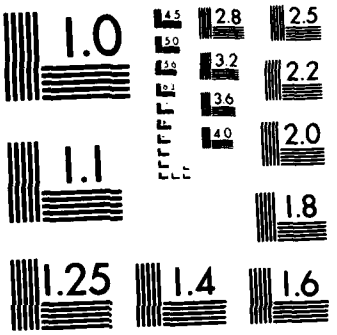

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TECHNICAL REPORT BRL-TR-2604

MULTIPHOTON PHOTOCHEMICAL AND COLLISIONAL EFFECTS DURING OXYGEN ATOM FLAME DETECTION

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October 1984

US ARMY BALLISTIC RESEARCH LABORATORY
ABERDEEN PROVING GROUND, MARYLAND

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A Nd:YAG pumped dye laser system was used to two-photon excite oxygen atoms at 225.6 nm in an atmospheric pressure $CH_4/N_2O/N_2$ flame. Subsequent emission at 844.7 nm from the directly populated state, as well as a stronger emission at 777.5 nm due to the $O(3p\ ^3P \rightarrow 3p\ ^5P)$ collisional energy transfer process, was monitored. Two-photon resonant oxygen atom and hydrogen atom (656.3 nm) emissions were also observed in the absence of a flame. Closer examination revealed that the tightly focussed probe beam was producing

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these atoms by promoting multiphoton photolysis of the oxidizer, as well as fuel molecules. Thus, this type of laser diagnostic probe is potentially quite intrusive depending on the combustion region that is probed, as well as the laser energies used.

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I. INTRODUCTION

Propellant combustion is a process of considerable complexity in which high temperature chemical reactions play a major role. In order to understand the actual heat releasing chemical steps that are involved, an extensive program has been ongoing at BRL, as well as in other combustion research institutions, where much simpler laboratory model flames are studied in detail. Such studies involve the interplay of flame diagnostics (usually based on laser spectroscopy techniques) with flame modeling work, as well as allied kinetics efforts to supply necessary elementary reaction rate constants for such detailed chemical flame models. In the area of flame diagnostics, laser spectroscopy techniques such as absorption, Raman, laser induced fluorescence (LIF), coherent anti-Stokes Raman spectroscopy (CARS), etc., have been able to detect the stable major flame species as well as many reactive molecular intermediates. There is, however, an important class of combustion intermediates, i.e., the flame atoms, such as H, O, and N, which has historically eluded direct detection due to the lack of accessible energy levels for single photon excitation.

Very recently, though, multiphoton excitation techniques have been successful in detecting the hydrogen and oxygen atoms in a flame. So far, two distinct detection approaches have been demonstrated, one involving multiphoton emission (MPE)[1,2] and the other multiphoton ionization (MPI),[3,4,5] the latter also referred to as optogalvanic detection. In the case of the oxygen atoms, however, direct detection in a flame was first accomplished by spontaneous Raman scattering[6] and coherent anti-Stokes Raman spectroscopy (CARS).[7] Apparently, sensitivity limits and spectral interferences have prevented further development of these spectroscopies for atomic flame detection.

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[1] *M. Alden, H. Edner, P. Grafstrom, and S. Svanberg, "Two-Photon Excitation of Atomic Oxygen in a Flame," Opt. Comm., Vol. 42, p. 244, 1982.*

[2] *R.P. Lucht, J.T. Salmon, G.B. King, D.W. Sweeney, and N.M. Laurendeau, "Two-Photon-Excited Fluorescence Measurement of Hydrogen Atoms in Flames," Opt. Lett., Vol. 8, p. 365, 1983.*

[3] *J.E.M. Goldsmith, "Resonant Multiphoton Optogalvanic Detection of Atomic Hydrogen in Flames," Opt. Lett., Vol 7, p. 437, 1982.*

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[5] *P.J.H. Tjossem and T.A. Cool, "Detection of Atomic Hydrogen in Flames by Resonance Four-Photon Ionization at 365 nm," Chem. Phys. Lett., Vol. 100, p. 479, 1983.*

[6] *C.J. Dasch and J.H. Bechtel, "Spontaneous Raman Scattering by Ground-State Oxygen Atoms," Opt. Lett., Vol. 6, p. 36, 1981.*

[7] *R.E. Teets and J.H. Bechtel, "Coherent Anti-Stokes Raman Spectra of Oxygen Atoms in Flames," Opt. Lett., Vol. 6, p. 458, 1981.*

Both the MPI and the MPE approaches suffer certain limitations: MPE always has quenching considerations as well as photometric inefficiencies, while MPI lacks selectivity in signal detection, and is thus quite susceptible to background ionization. In either approach, the nonlinear signals which usually depend on the number of photons, n, required for the given transition, are generally low due to the characteristically small multiphoton absorption cross sections. Thus, tight focusing and maximum laser energy extraction is typically desirable. We have made recent observations in our laboratory, however, which suggest that at sufficiently high laser energies, the flame diagnostic probe can become substantially intrusive by promoting multiphoton dissociation of the parent fuel and oxidizer molecules, in this case into hydrogen and oxygen atoms. Also, we have observed very efficient collisional energy transfer from the two-photon populated oxygen level to a neighboring level, as well as energy transfer from the excited oxygen to hydrogen atoms (see Figure 1). The two-photon excitation scheme for oxygen atoms using 225 nm photons, followed by detection of emission at 845 nm (see Figure 1), was initially demonstrated in a low pressure discharge[8] and then applied successfully for the first time to a combustion environment in an experiment involving the C_2H_2/O_2 flame.[1]

II. EXPERIMENTAL

Figure 2 shows the experimental schematic. A Nd:YAG laser is used to pump a dye laser in which R590 dye is circulated. The output is frequency doubled and mixed with the 1.06 micron Nd:YAG fundamental in a commercial wavelength extender to produce tunable radiation in the 225 nm range. This beam is then focussed with a 100 mm focal length lens into a curved knife-edge burner, whose edges are separated by 3 mm from each other, and in which a fuel rich $CH_4/N_2O/N_2$ flame (0.14:0.38:0.48) is stabilized. Laser pulse widths of 5 nsec and energies up to 1.5 mjoules/pulse at 10 pps were employed. The emitted radiation from the flame was collected by a lens system and passed through a broadband filter into a 0.3 m monochromator whose slits were relatively wide (ca. 2 nm) and finally detected by a photomultiplier tube (EMI 9659QA). The PMT output was fed into a 7912AD Tektronix transient digitizer, which was interfaced with a PDP 11/04 computer. Typically 50-300 laser shots were averaged per data point.

III. RESULTS AND DISCUSSION

Figure 3 shows an O atom emission profile through the curved knife-edge burner. A well-defined flame front is quite evident while the second peak is interpreted as resulting from entrained air further reacting with the fuel-rich post-flame gases. Figure 3 is based on monitoring the 777.5 nm line, which is actually a grouping of three closely spaced lines originating from the three ^5P upper states to the common $^5S_2^o$ state. The emission intensity of this group as measured in the primary flame zone was about an order of

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[8] *W.K. Bischel, B.E. Perry, and D.R. Crosley, "Two-Photon Laser-Induced Fluorescence in Oxygen and Nitrogen Atoms," Chem. Phys. Lett., Vol. 82, p. 85, 1981.*

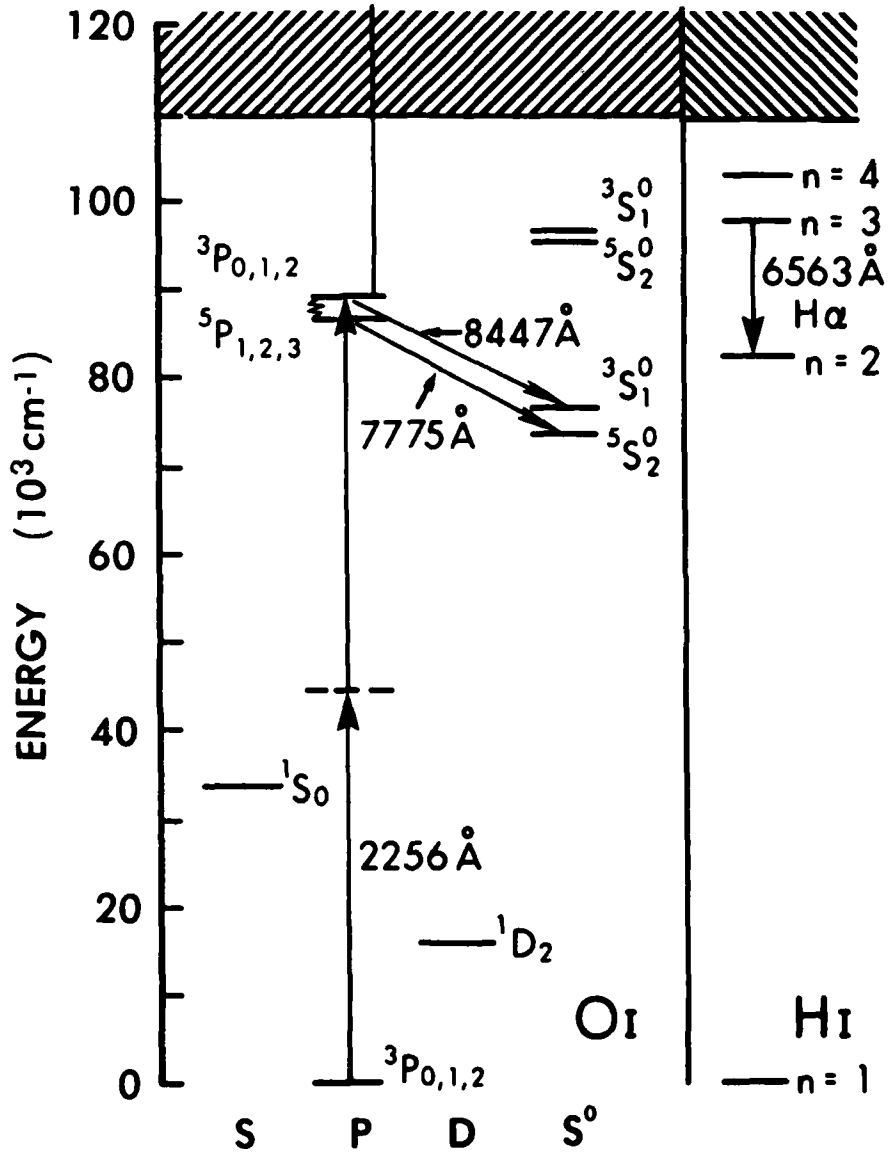

Figure 1. Partial Energy Level Diagram for O and H Atoms

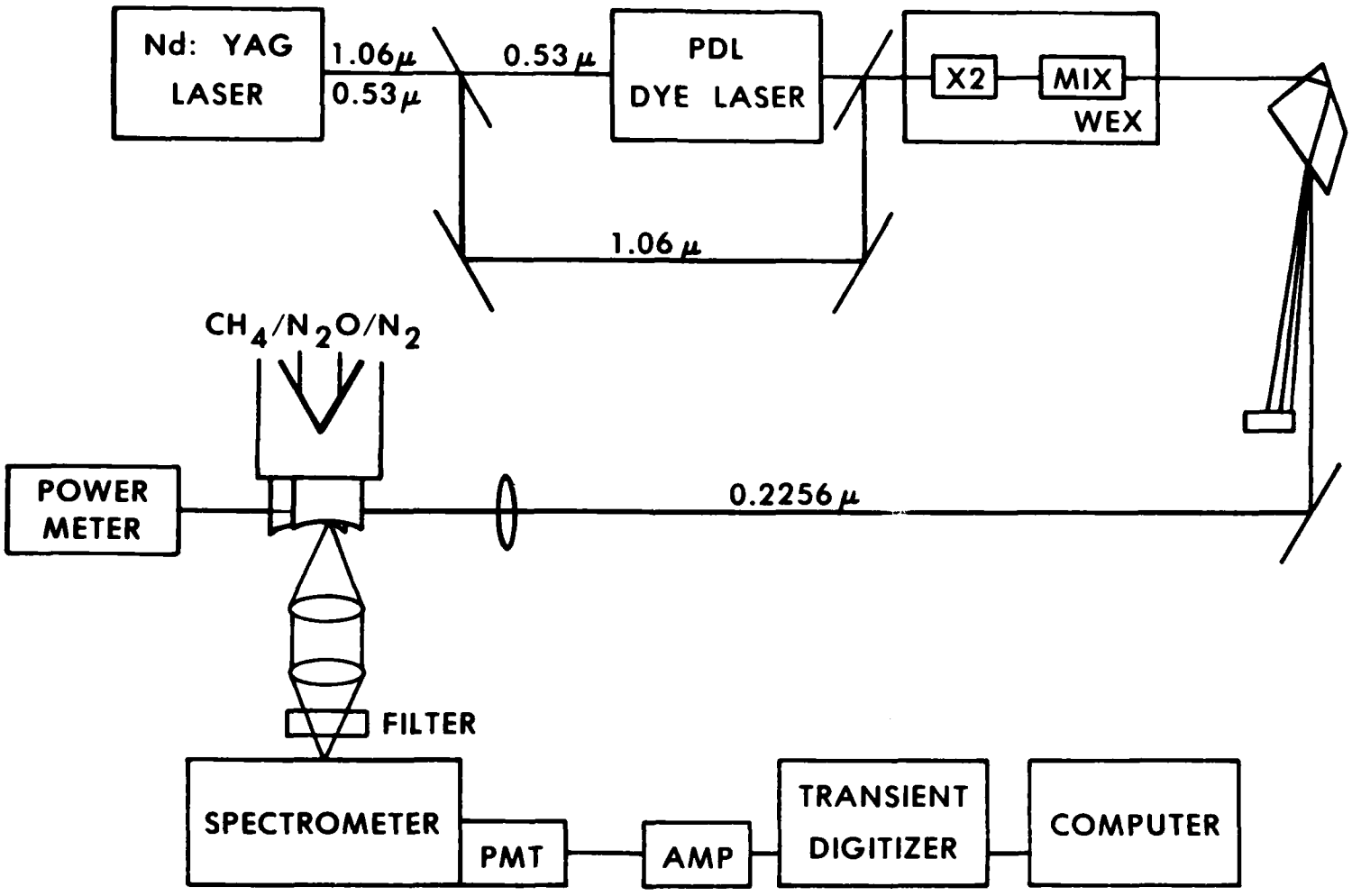

Figure 2. Experimental Schematic for O-Atom MPE Experiments

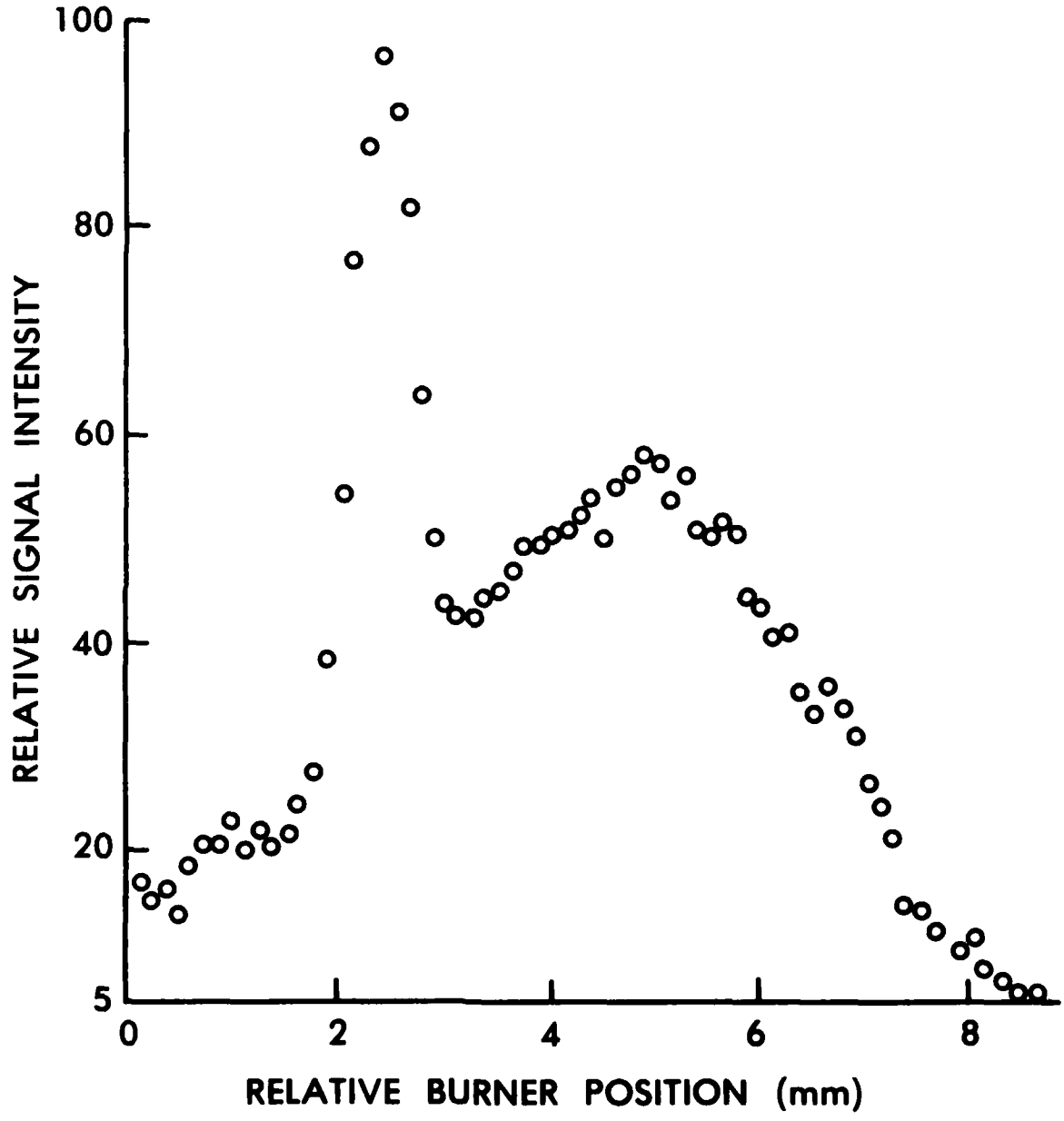

Figure 3. O Atom Emission Profile at 777.5 nm in a Curved Knife-Edge Burner Supporting a Fuel-Rich $CH_4/N_2O/N_2$ Flame

magnitude greater than the emission at 844.7 nm. When photometric wavelength differences, such as grating and PMT quantum efficiencies are taken into account, the 777.5/844.7 emission ratio is still about 3. This indicates an efficient energy transfer process and has desirable analytical implications since now two different wavelengths are available for O atom detection. Due to the fact that low signal levels were usually a problem, most of the experiments were done monitoring the 777.5 nm line.

Figure 4 shows the O atom two-photon excitation spectra where the ground spin-orbit states are resolved. The observed ratio of 9.5:4.1:1 can be compared to a Boltzmann calculated ratio of 5.8:3.1:1 for an adiabatic flame temperature of 2290°K. This, in turn, can be compared to the ratio of 1.6:1.3:1, which was observed in optogalvanic experiments on the $H_2/O_2/Ar$ flame.[4] The reason for these discrepancies is not clear at this time, but recently there have been further theoretical efforts to substantiate the two-photon absorption cross sections which were found to be equal for each of the three fine-structure components involved in this transition.[9] A rough estimate of 0.0001% for the detection limit is based on the emission intensity in the primary flame zone (see Figure 3) and a calculated O atom mole fraction of 0.005% (NASA/Lewis equilibrium program). This number should be improved by at least an order of magnitude by optimizing the optical collection efficiency, as well as using a red-sensitive PMT. This detection limit can be compared to 0.01% indicated in the previous MPE experiment,[1] parts-per-million sensitivity for the MPI experiment,[4] as well as 0.01-0.1% for the two Raman experiments.[6,7]

Further spectral investigations revealed another emission line centered at 656.3 nm, which was generally as intense as the 777.5 nm line and was resonant with O atom two-photon excitation. We have identified this emission as the H_α transition in the hydrogen atom Balmer series (see Figure 1). However, the mechanism for populating the n=3 level, which is 8861 cm^{-1} higher in energy than the laser populated O atom level, was quite unclear for some time. Another even more surprising observation was the detection of the two O atom emission lines, as well as the 656.3 nm H-atom line in the absence of a flame when only room temperature premixed gases were flowing through the burner. In order to understand these findings, a determination of the order of nonlinearity, n, of these signals under different conditions was initiated and the results are given in Table 1. The uncertainties for the value of n are based on a number of different runs in which the Nd:YAG amplifier lamp energy was varied to yield energies between 0.2-1.5 mjoule/pulse. Clearly, this is not the best way to vary the probe laser energy since the output pulse time profile may change somewhat. Unfortunately, intensity attenuation attempts using polarizers available in our laboratory were unsuccessful since the coatings could not withstand the high peak intensities and short wavelengths used. Nevertheless, the results listed in Table 1 are consistent with a qualitative picture of multiphoton induced photolysis of the fuel and oxidizer parent molecules followed by a O atom two-photon resonant formation of a microplasma. In the preheat region the value of n=4 is close to what is found for the N_2O room temperature flow where n=5, suggesting that multiphoton

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[9] *D.R. Crosley and W.K. Bischel, "On Relative Fine-Structure Intensities in Two-Photon Excitation," Phys. Rev., in press 1984.*

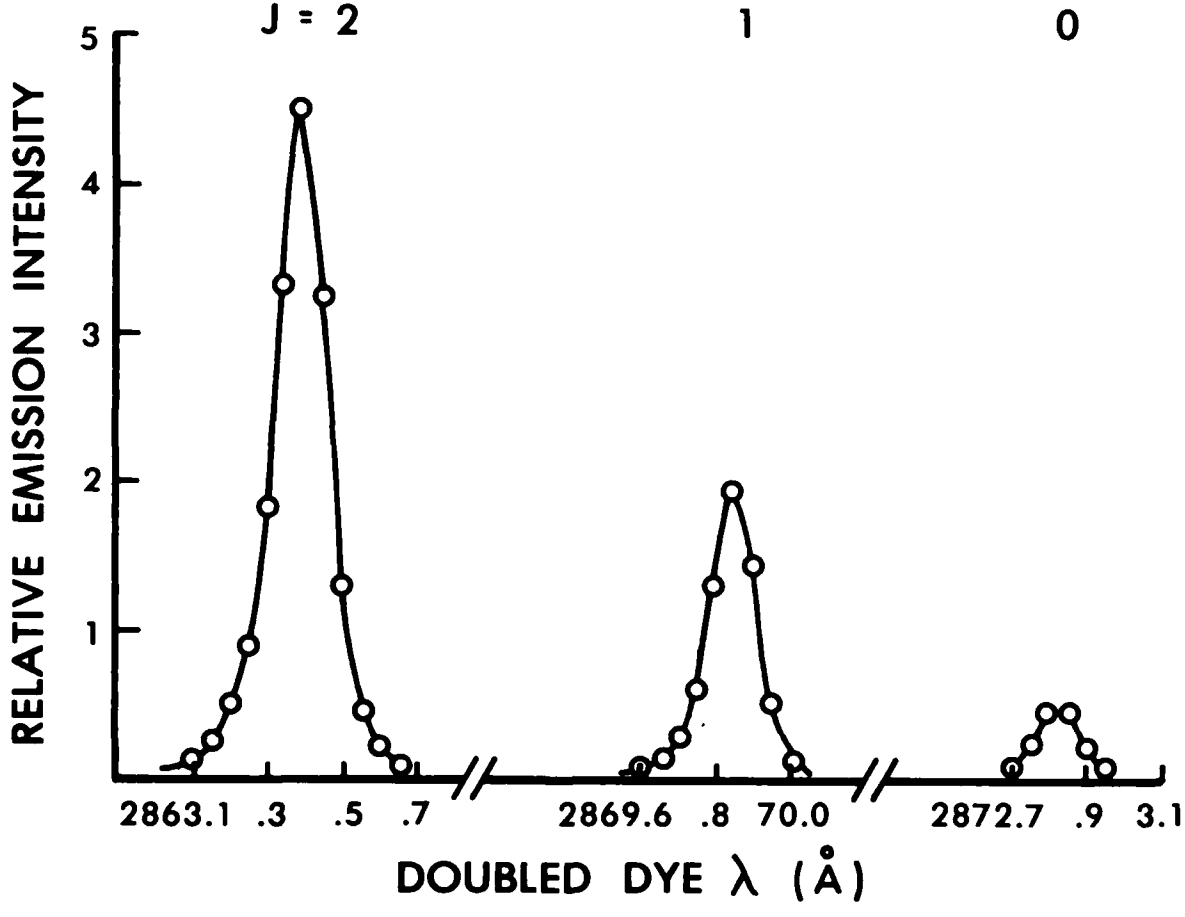

Figure 4. O-Atom Emission at 777.5 nm Due to Spin-Orbit Resolved Ground States in the Primary Flame Zone

TABLE 1. ORDER OF NONLINEARITY (n) OF MULTIPHOTON INDUCED EMISSION SIGNALS[a]

Atom (Emission λ)	Flame Region Probed	n
O (777.5 nm)	Preheat	4 ± 0.5
"	Primary	2 ± 0.5
"	Post-Flame	1.8 ± 0.5
"	Room Temp. Flow	5 ± 1
H (656.3 nm)	"	10 +

a - Where emission signal α (laser energy)^n

photolysis of N_2O is contributing significantly to the total O atom emission signal. In the primary and post-flame regions, however, the value of n=2 is just what one would expect for a two-photon generated signal where the photolytic parent, N_2O, has already reacted away and the native O atom flame population is relatively high. The highest order of nonlinearity is found for the H atom emission which is expected since it takes some number of photons to photolyze CH_4, and more for N_2O, and finally two to excite the O atoms. The lack of uncertainty limits for the H-atom case attests to the difficulty (i.e., long averaging times and high degree of scatter) involved in making measurements on very high orders of nonlinearity.

A microplasma is not surprising since the absorption of the third photon ionizes the O atoms[4] and the temperature in the focal volume may be much hotter than even the flame temperature, thus promoting collisional energy transfer from O to H atoms. An alternative to multiphoton photolysis might be plasma chemistry involving highly excited states or ion-molecule reactions, but these explanations seem less likely since all of the emissions are prompt, i.e., 5 nsec or less apart from each other. Again, it should be stressed that the results in Table 1 are qualitative in nature since signal levels, especially where n is high, were quite low, requiring laser energies over 1 mjoule/pulse, and they are based on the collisionally induced 777.5 nm line where the rates of collisional energy transfer may not be constant for the different experimental conditions. Clearly, this is a complex situation which deserves further study. Nevertheless, the potential for the multiphoton diagnostic probe to be intrusive certainly exists and thus appropriate caution should be exercised in these types of experiments. The implication of our results to previous multiphoton O atom flame studies cannot be readily assessed since those were done on flame systems other than the $CH_4/N_2O/N_2$ and thus photochemical effects (if any) are probably quite different. Finally, multiphoton induced photolysis should, in fact, not be too surprising since recent literature in chemical physics has many examples of such, even

involving typical flame molecules like H_2[10] or C_2H_2.[11] There is even a recent flame paper where laser probe intrusion (presumably photochemical) was noted.[12] On the other hand, as pointed out by those authors, there may be new opportunities to expand our knowledge of combustion by exploiting photochemistry.

IV. CONCLUSIONS

The following conclusions can be reached on the basis of our experiments.

1. Oxygen atoms can be detected in a $CH_4/N_2O/N_2$ flame by two-photon excitation at 225.6 nm and emission detection at 844.7 nm.

2. The collisional energy transfer between the directly populated ^3P excited oxygen atom state and the neighboring ^5P state is very efficient. This is quite surprising since the process involves the change of spin multiplicities. For a given experimental condition, the resulting emission at 777.5 nm can be much more intense than the one at 844.7 nm and thus be more useful for sensitive analysis of O-atoms.

3. Hydrogen atom emission at 656.3 nm is detected, which is resonant with O atom two-photon excitation. This may result from an endothermic collisional energy transfer process occurring in the microplasma formed in the focal volume of the laser whose temperature is hotter than the flame temperature. Alternative explanations, however, for the H-atom emission such as plasma chemistry/fast ion-molecule reactions, cannot be ruled out at this time.

4. Since both oxygen atom and the single hydrogen atom emissions were detected without a flame, the multiphoton photolysis of the atom precursors, N_2O and CH_4, was indicated. This was substantiated by a study of the order of nonlinearity of the emission signal.

5. The above observation indicates that the laser multiphoton flame diagnostic probe is potentially quite intrusive, depending on the region of the flame being studied (i.e., the relative concentration of photochemical precursors), as well as the laser energies utilized (i.e., the higher the laser energy, the more prominent the higher order processes become). Thus, appropriate caution should be exercised in these kinds of experiments.

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[10] *S.T. Pratt, P.M. Dehmer, and J.L. Dehmer, "Resonant Multiphoton Ionization of H_2 via the $B^1\Sigma_u^+$, v=7, J=2, and 4 Levels with Photoelectron Energy Analysis," J. Chem. Phys., Vol. 78, p. 4315, 1983.*

[11] *B.B. Craig, W.L. Faust, L.S. Goldberg, and R.G. Weiss, "UV Short-Pulse Fragmentation of Isotopically Labeled Acetylene: Studies of Emission with Subnanosecond Resolution," J. Chem. Phys., Vol. 76, p. 5014, 1982.*

[12] *M. Alden, H. Edner, and S. Svanberg, "Simultaneous Spatially Resolved Monitoring of C_2 and OH in a C_2H_2/O_2 Flame Using a Diode Array Detector," Appl. Phys. B, Vol. 29, p. 93, 1982.*

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